भारतीय मानक
Indian Standard

IS 11552 : 2020

75 लीटर तक की क्षमता के द्रव नाइट्रोजन आधान — विशिष्टि

(दूसरा पुनरीक्षण)

Liquid Nitrogen Vessels of Capacity up to 75 Litres — Specification

(Second Revision)

ICS 23.020.01

भारतीय मानक ब्यूरो
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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Chemical Engineering Plants and Related Equipment Sectional Committee, had been approved by the Mechanical Engineering Divisional Council.

This standard was first published in 1986 and subsequently revised in 2008. Since then many suggestions were received for improvement and Amendment No. 1 and 2 was issued. This standard is being revised again to incorporate the amendment issued and the suggestions received from time to time.

The liquid nitrogen vessels are suitable for storage and transportation of liquid nitrogen in liquid form. These vessels are also used for preserving various materials under frozen conditions without any change in their characteristics. Such vessels are normally used in scientific research work, such as cryosurgery, space research artificial insemination processes, embryonic stem cells, etc, and shrink fitting applications.

The Composition of the Committee responsible for the formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Indian Standard

LIQUID NITROGEN VESSELS OF CAPACITY UP TO 75 LITRES — SPECIFICATION

(Second Revision)

1 SCOPE

This standard covers liquid nitrogen vessels that are double walled with multilayer, vacuum insulated construction and of capacities up to 75 litres for storage and transportation of liquefied nitrogen. These vessels are also termed vacuum vessels or cryo refrigerators or liquid nitrogen containers. The standard lays down requirement of construction and testing of above vessels. These arc non-pressurized and non-explosive vessels.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
737 : 2008	Wrought aluminium and aluminium alloy sheet and strip for general engineering purposes (*third revision*)
2500 (Part 1) : 2000	Sampling inspection procedure: Part 1 Attribute sampling plans indexed by acceptable quality level (AQL) for lot by lot inspection (*third revision*)
6911 : 2017	Stainless steel plate, sheet and strip — Specification (*second revision*)
7028 (Part 2) : 2002	Performance tests for complete, filled transport packages: Part 2 Vibration test at fixed low frequency (*second revision*)
14811 : 2000	Rolled copper plate, sheet, strip and foils for general engineering purposes — Specification

3 GENERAL REQUIREMENTS

3.1 Material of Construction

3.1.1 The vessels and the dispenser unit shall be manufactured either with austenitic stainless steel conforming to IS 6911 or aluminium or its alloys conforming to IS 737 or copper or its alloys conforming to IS 14811.

3.1.2 Openings like inlet, pump out-port etc, and supports or stays shall be manufactured out of material suitably selected, so that the losses due to conductive heat transfer through such components is restricted to the minimum.

3.1.3 Fibre glass components, where used, shall be so manufactured that the finished components meet the requirements under vacuum as well as cryogenic conditions/applications.

3.2 Construction

3.2.1 The vessel shall be insulated by using super insulation/multilayer insulation technique. The insulation material used shall have good resistance against conductive and radiative heat transfer The minimum thickness for the inner and outer shell shall be 1.2 mm for aluminium or its alloy, 1.6 mm for austenitic stainless steel, and 1.2 mm for copper or its alloys.

3.2.2 The annular space between inner and outer shell may vary depending on the super insulation technique adopted.

3.2.3 The annular space shall be evacuated and the vacuum level shall be 0.133 Pa (10^{-3} torr) when measured in pumping header at ambient temperature. The vacuum space in the vessel shall be sealed off at such vacuum.

3.2.4 The bond between the inner and outer shell shall be suitable for vacuum and cryogenic applications.

3.2.5 The annular space shall be provided with appropriate and adequate quantity of adsorbent material to ensure maintenance of quality vacuum.

3.2.6 A pump out-port shall be provided on the body of the outer container at suitable location with suitable vacuum seal and a protective cap. Outer container shall be provided with suitable device to release internal pressure.

3.2.7 The welding of the joints shall be done by TIG/MIG (GTAW/GMAW) welding process only.

3.2.8 Vessels up to and including 12 litres shall be provided with portable handles which may or may not be removable for ease of movement and lifting. For models above 12 litres shall be provided with the handles at convenient location. For models of capacity 2 litres and below, handle is optional and such models may be supplied with Rexene/canvass bag.

3.2.9 No permanent cap or sealing device shall be provided to close the inner vessel unless it has breathing or pressure release device provided to release excessive internal pressure.

3.3 Specific Requirements

Vessels shall conform to the requirements specified in **10** and **11** as applicable.

4 TESTING

4.1 Purpose

The quality of vessel is determined by the rate of evaporation at Standard Temperature and Pressure (STP) of the cryogenic liquid contained in it without canisters. Test procedure shall therefore be oriented to find the liquid loss under site conditions and converting the value to STP condition. The loss rate shall be determined as per stabilization time declared by the manufacturer.

4.2 Performance Test

4.2.1 The vessel shall be filled with liquid nitrogen and the minimum quantity to be filled is 20 percent of its capacity at the time of first weighment and contents shall be stabilized for a minimum period of 12 h before taking the first weight.

4.2.2 After stabilization, the weights, date, time and average temperature shall be recorded. The average temperature shall be based on minimum and maximum temperature recorded during the period of 24 h.

4.2.3 The weighments shall be done on the electronic balance with 5 g least count or better.

4.2.4 Weighments shall be done at interval of 24 ± 1 h or its multiples. The vessel shall be fitted with its neck plug and without canisters during the course of test. This shall be done for a minimum of two weight differences and the last weight difference shall be taken as the loss rate and shall be recorded.

4.2.5 The rate of evaporation at site conditions shall be converted to STP condition by adopting formula of ideal gasses:

$$\frac{P_1 V_1}{T_1} = \frac{P_2 V_2}{T_2}$$

where

P_1 = atmospheric pressure at the testing site, in mm;
P_2 = 760 mm;
V_1 = loss in litres, under test conditions;
V_2 = loss in litres, at STP;
T_1 = temperature during test, in kelvin; and
T_2 = 273 in kelvin.

Manufacturer has to establish their own documented procedure for estimation of loss rate. A guideline for calculation of loss rate at 15°C is given in Annex A.

4.2.6 The evaporation loss of the vessel shall not be more than the limits specified in Table 1 and Table 2 at STP, as applicable.

Table 1 Evaporation Losses of Refrigeration Vessels

(*Clauses* 4.2.6 *and* 10.3)

Sl No.	Capacity of Vessel	Evaporation Loss Allowed in Litres per Day Refrigeration Vessels		
	Liters	For FRP Neck Up to 60 mm Diameter	For FRP Neck 61 to 100 mm Diameter	For FRP Neck 101 to 150 mm Diameter
(1)	(2)	(3)	(4)	(5)
i)	Up to 12	0.15	0.30	0.40
ii)	Over 12 up to 35	0.13	0.40	0.60
iii)	Over 35 up to 45	0.40	0.60	0.80
iv)	Over 45 up to 75	0.50	0.75	1.00

Table 2 Evaporation Losses of Transportation Vessels

(*Clauses* 4.2.6 *and* 11.3)

Sl No.	Capacity of Vessel	Evaporation Loss Allowed in Litres per Day Transportation Vessels		
	Liters	For FRP Neck Up to 60 mm Diameter	For FRP Neck 61 to 100 mm Diameter	For FRP Neck 101 to 150 mm Diameter
(1)	(2)	(3)	(4)	(5)
i)	Up to 12	0.20	0.30	0.40
ii)	Over 12 up to 25	0.25	0.40	0.60
iii)	Over 25 up to 35	0.30	0.50	0.75
iv)	Over 35 up to 50	0.50	0.75	1.25
v)	Over 50 up to 75	0.60	1.00	1.50

4.2.7 Derived tests and SQC techniques will be suitably established, documented and used for the evaluation and for performance testing of rates of evaporation of vessels. Sampling shall be as per Table 1, General inspection level II and IIA of IS 2500 (Part 1) for AQL level 2.5 for all models.

4.2.8 If the sample is rejected, performance testing of 100 percent shall be done by liquid nitrogen for the complete batch.

4.2.9 *Leak Tightness Test*

The complete outer vessel shall be Mass Spectrometer Leak Detector (MSLD) tested in the order of 1×10^{-8} Pa m^3 /s (1×10^{-7} mbar 1/s). MSLD testing shall be carried out as laid down in Annex B.

4.3 Type Test

Vibration test followed by performance test shall be carried out in accordance with **10.4** or **11.4** as applicable.

5 CAPACITY OF THE VESSEL AND CHANGE IN DESIGN OF THE VESSEL

5.1 Water Capacity

5.1.1 *Storage, Transport and Refrigerated Vessels*

The capacity of the vessel shall be determined from the water volume of the inner vessel, up to the bottom level of the neck tube.

5.1.2 The tolerance for water capacity shall be ± 2 percent.

5.2 Change of Design of Vessel

5.2.1 The following shall constitute the change in the design of vessel:

a) Any change of the type/grade of material than the present used one;

b) Any change or increase in fundamental shape of the vessel and Fiber Reinforced Plastic (FRP) neck; and

c) Increase in nominal length or diameter of the inner shell, that is more than 10 percent of the present vessel.

5.2.2 In event of any of the above changes, all tests as laid down in this standard shall be repeated.

6 MARKING

6.1 The liquid nitrogen vessel shall bear the following markings in legible and durable characters on the outer shell of the vessel:

a) IS 11552 to show that the vessel is in conformity to this standard;

b) Liquid nitrogen (in bold letters);

c) Nominal volume of inner vessel, in litres;

d) Serial number of vessel and year of manufacture; and

e) Name and address or other means of identification of manufacturers.

6.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

7 PAINTING

Painting of the vessel is for improving the aesthetics. If employed then epoxy or polyurethane paint, which is suitable for cryogenic applications, shall be used for painting.

8 INSTRUCTIONS FOR USE AND SAFETY PRECAUTIONS

Each liquid nitrogen vessel shall be accompanied with a bilingual (one language shall be English) guide/manual which shall include, in particular, at least the following:

a) Introduction of the equipment;

b) Instructions about the dangers related to liquid nitrogen. It shall contain at least the following:

   1) Liquid nitrogen is a very cold fluid having extremely low temperature of -196°C;

   2) Liquid nitrogen when vapourizes, occupies large volumes, can displace oxygen causing asphyxiation; and

   3) If spilled on body parts, can cause severe 'cold burns'.

c) Instructions related to handling of the equipment; and

d) Instructions about safety precautions to be taken while handling liquid nitrogen. It shall contain at least the following:

   1) Wear protective gear comprising of insulated hand gloves (preferably loose), protective goggles and safety shoes while handling liquid nitrogen;

   2) Keep vessel in well ventilated area;

   3) Keep clear of splashing and boiling liquid nitrogen;

   4) Use correct equipment/proper accessories for liquid nitrogen transfer;

   5) While inserting warm (room temp) objects into the vessel filled with liquid nitrogen, do it gradually to avoid sudden boil-off;

   6) In case of spillage on body parts, wash with large quantity of room temperature water as first aid as well as call doctor. Do not rub frozen parts as tissue damage may result and never use dry heat;

   7) In case of as phyxiation, immediately call doctor with oxygen facility, in the meanwhile shift affected person in well ventilated place as first aid; and

   8) Use only the accessories supplied with the equipment.

9 PACKING

All liquid nitrogen vessels should be packed in corrugated boxes and the ply size suitable for the rail/ road conditions. The serial number and model should be written on the corrugated boxes. Other forms of packaging may also be employed as agreed between the manufacturer and the buyer.

10 REQUIREMENTS APPLICABLE TO LIQUID NITROGEN VESSELS FOR REFRIGERATION/ STORAGE APPLICATIONS

10.1 The refrigeration-cum-storage vessels shall have a dividing device/locating device for the vessels up to and including 12 litres which can be either at top near the neck or at bottom inside the container. For capacities above 12 litres to have dividing devices at both the location, that is, at the bottom of the inner vessel and also at the neck, for easy insertion, undisturbed location and smooth removal of the canisters.

10.2 They shall be provided with stoppers made out of suitable foam material for cryogenic application, rigidly fixed to the top caps. They shall have suitable grooves to accommodate canisters.

10.3 The evaporation loss of such vessels shall be restricted to the limits as specified in Table 1 at standard temperature and pressure conditions after specified tests. Depending upon neck diameter and capacity, the loss rate may vary. Under such case, the manufacturer shall declare the loss rate.

10.4 For any new models, after assessing the performance in accordance with **4.2** and vibration test in accordance with **10.4.1** performance test shall be carried out again in accordance with **10.4.2.**

10.4.1 Three vessels shall be subjected to vibration test with amplitude of 25 ± 0.5 mm in normal packed condition (without accessories) without liquid nitrogen to an acceleration of 1 g for 1 h. The vibration test shall be carried out according to method specified in IS 7028 (Part 2).

10.4.2 After vibration tests the vessels shall be again subjected to performance test laid down in **4.2** for seven days for vessel having capacity more than 1 liters and 3 days for vessels having capacity less than 1 litres and a deviation of 25 percent from the initial loss rate is acceptable. If one or more of these three vessels are not found satisfactory in performance testing, a fresh batch of ten vessels shall be manufactured and tested as above and all of them shall meet the requirements. The duration of performance test after vibration test shall be maximum 30 days.

11 REQUIREMENTS APPLICABLE TO LIQUID NITROGEN VESSELS FOR TRANSPORT APPLICATIONS

11.1 The liquid nitrogen vessels of capacities greater than 25 litres for transport applications shall be designed for meeting the road conditions with proper supports or stays.

11.2 They shall be provided with stoppers made out of suitable foam material, rigidly fixed to the top caps.

11.3 The evaporation loss of such vessels shall be restricted to the following limits specified in Table 2 at standard temperature and pressure conditions after specified tests. Depending upon neck diameter and capacity the loss rate may vary, under such a case the manufacturer shall declare the loss rate.

11.4 For any new models, after assessing the performance in accordance with **4.2** and vibration test in accordance with **11.4.1** performance test shall be carried out again in accordance with **11.4.2**.

11.4.1 Three vessels shall be subjected to vibration test with amplitude of 25 ± 0.5 mm in normal unpacked condition (without accessories but with rubber bottom pad) without liquid nitrogen to an acceleration of 1 g for 1 h and with liquid nitrogen to an acceleration of 1 g for 1 h. The vibration test shall be carried out according to method specified in IS 7028 (Part 2).

11.4.2 After vibration tests the vessels shall be again subjected to performance test laid down in **4.2** for seven days for vessel having capacity more than 1 liters and 3 days for vessels having capacity less than 1 litres and a deviation of 25 percent from the initial loss rate is acceptable. If these three vessels are found satisfactory in performance testing, a fresh batch of ten vessels shall be manufactured and tested as above. The duration of performance test after vibration test shall be maximum 30 days.

11.5 All the transport models of capacities greater than 25 litres shall be fitted externally with a rubber bottom pad and at least two numbers of side rubber rings.

12 ACCESSORIES

12.1 Accessories, if supplied, shall be in accordance with the requirements laid down in **12.2** to **12.4.**

12.2 Manual and Automatic Dispenser for Transferring the Liquid Nitrogen

12.2.1 This is a combination of a standard liquid nitrogen vessel and an automatic dispenser head which can be attached together as and when required.

12.2.2 The dispenser head shall be made out of material like aluminum alloy material and/or stainless steel that is suitable for liquid nitrogen service, wetted parts only.

12.2.3 All joints of the dispenser head shall be suitably tested for leakage by employing air/nitrogen at a pressure of 50 kPa (gauge) and all screwed, welded and brazed joints to be similarly tested at a pressure of 80 kPa (gauge). There shall be no leakage at the above test pressures.

12.2.4 The neck of the vessel and the dispenser head shall be provided with suitable flanges and seal ring or silicon rubber bush, fitted to dispenser unit suitable for neck ID of vessel. The seal ring or silicon rubber bush shall be of shore hardness 40 to 45 on scale A. The dispenser shall be provided with suitable arrangement for clamping it on the vessel.

12.2.5 All welding shall be done by TIG/MIG (GTAW/ GMAW) process.

12.2.6 The dispenser head shall have the following fittings:

a) A delivery line with suitable valve to ensure controlled flow of liquid;

b) A nipple or valve for external pressurization or for tapping of low pressure gas or for emergency pressure release;

c) A spring loaded pressure relief valve shall be provided. The relief pressure shall be set at 49 to 54 kPa (gauge) and to be suitably sealed;

d) A suitable Bourdon type pressure gauge of range 0 to 98 kPa (gauge) may be provided; and

e) The dispenser should have additional fastening device like chain/clamps/straps which can be suitably tied to the handle of the liquid nitrogen vessels during usage.

12.2.7 The delivery rate of liquid nitrogen through the dispenser shall not be more than 4 l/min.

12.2.8 The automatic transfer device may be electrically operated with proper power source, pressure switch, heaters and relief valves for safe operation.

12.2.9 All dispenser and its parts shall be suitably packed as agreed between supplier and the purchaser.

12.3 Funnel

For easy transferring of liquid nitrogen use stainless steel funnel to prevent splashing and spilling. This funnel to have suitable supporting arrangement to avoid any direct contact with the FRP neck.

12.4 Trolleys

The handling and movement of liquid nitrogen vessels used for the transportation applications to be done through the handling trolleys designed with respect to the diameter of the vessels. The container can be easily mountable, detachable and movable. Proper rubberized wheels and necessary clamping arrangement of the container to be provided with cushioning effect.

ANNEX A

(*Clause* 4.2.5)

GUIDELINES FOR CALCULATION OF LOSS RATE AT 15° C

A-1 To calculate the average loss rate of liquid nitrogen per day, after the container is stabilized the difference in weight is to be obtained throughout the period. The loss rate is to be corrected to 15° C by using correction factor as given in table below.

Example:

First weight on first day :	A_1
Second weight on second day :	A_2
Third weight on third day :	A_3
First difference in weight =	A_1-A_2 = $W_1 g$ with an average temperature of 26° C
Second difference in weight =	A_2-A_3 = $W_2 g$ with an average temperature of 28°C

The loss rate at 150 is calculated by multiplying W_1 with corresponding multiplying factor at 26° and by multiplying W_2 with corresponding factor at 28°C.

Temperature Correction Factor Table

Temperature	Multiply by
°C	
0	1.263
1	1.243
2	1.223
3	1.204
4	1.185
5	1.166
6	1.148
7	1.130
8	1.113
9	1.096
10	1.079
11	1.063
12	1.047
13	1.031
14	1.015
15	1.000
16	0.984
17	0.970
18	0.956
19	0.942
20	0.928
21	0.914
22	0.900
23	0.887
24	0.874
25	0.862
26	0.849
27	0.837
28	0.825
29	0.813
30	0.802
31	0.790
32	0.779
33	0.768
34	0.757
35	0.747
36	0.735
37	0.725
38	0.717
39	0.708
40	0.698

Liquid Nitrogen density shall be taken as 0.808 kg/l.

The calculated loss rate of the container (for W_2 at temperature of 28°C) in terms of litres per day is = $W_2 \times 0.825/0.808$.

ANNEX B

(*Clause* 4.2.9)

HELIUM MASS SPECTROMETER LEAK DETECTOR

B-1 INTRODUCTION

The helium mass spectrometer is high sensitivity detector for detection of minute traces of helium gas, used for detecting minute leaks, either by pressurizing components with helium mixture or by evacuating the component and spraying helium from outside.

B-2 EQUIPMENT

B-2.1 Mass Spectrometer Leak Detector (MSLD)

B-2.2 Evacuation System

Rotary and roots pumping system with header, valves, instrument gauges for initial evacuation.

B-2.3 Calibration

The helium mass spectrometer is to be calibrated as per the instruments manufacturer's operation and maintenance manual using a permeation type leak standard to establish that the instrument is at optimum or adequate sensitivity of at least 1×10^{-10} Pa m^3/s (1×10^{-9} mbar l/s). Calibration shall be checked everyday before testing the components.

B-2.4 Ensure that all the sealing '*O*' rings are clean before connecting it to MSLD.

B-3 TESTING

B-3.1 Evacuate the inner vessel with vacuum level of 13.33 Pa (1×10^{-1} torr). Evacuate outer vessel after assembly with vacuum level of 66.67 Pa (5×10^{-1} torr) and then connect it to MSLD.

B-3.2 All the inner vessels shall be hood tested. For example, cover the complete inner vessel with a polythene bag and pass helium gas through small aperture to form a balloon and hold for at least 1 min.

B-3.3 All the outer vessels after assembly shall be tested by the help of the helium detector probe tip which is passed over the joints (welded as well as bonded) and also purging the inner vessel for 10 s. Alternatively, gross leak first may be checked by Hood technique and in case of any leak, it can be detected as above.

B-3.4 The leak tightness of inner vessels to be of order of 1×10^{-9} Pa m^3/s (1×10^{-8} mbar 1 /s) and outer complete vessels to be of order of 1×10^{-8} Pa m^3/s (1×10^{-7} mbar l/s).

B-3.5 When unacceptable leakage is detected, the location of leaks shall be marked. The vessels shall be repaired and after repairing the vessels shall be retested.

ANNEX C

COMMITTEE COMPOSITION

Chemical Engineering Plant and Related Equipment Sectional Committee, MED 17

Organization	*Representative(s)*
Indian Institute of Petroleum (IIP), Dehradun	Shri Amar Kumar Jain (***Chairman***)
Advance Valves Global, Noida	Shri Chandrakant Wadekar Shri Pranay S. Garg (*Alternate*)
Auma India Private Limited, Bengaluru	Shri Yashwant M. Jannu
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Organization	*Representative(s)*
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VAG-Valves (India) Pvt Ltd, Medak	Shri G. Satyanarayana

Member secretary

Shri Sandeep Keshav
Scientist 'B' (MED), BIS

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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